

Deepwater Horizon – Data Validation Report P.I.A.N.O. Volatile Organic Compounds

SDG: 1005011	Matrix: Water	Number of Samples: 15
Laboratory: Alpha Analytical Services		Method/SOP: Alpha VOC_Piano SOP/0-019 (Issue 2)
Validation Level: Stage 2B Validation		Validation Criteria Table: MC252-VOC, Rev. 0
Date of Report: 6/21/2010		Approved for Release:

Refer to **ATTACHMENT 1: SAMPLE INDEX** for a list of validated samples.

Refer to the **DATA VALIDATION PLAN** for validation approach, Criteria Tables, qualifier and reason code definitions.

The quality control (QC) elements that were reviewed are listed below.

√	Data Package Completeness	√	Sample Duplicate Analysis
√	Verification of EDD to Hardcopy Data Package	√	Blank Spike/Blank Spike Duplicate Sample Analyses
√	Chain-of-Custody and Sample Receipt	2	Reference Material Analysis
√	Holding Times	√	Internal Standards
√	Instrument Tuning	√	Detection Limits
1	Initial Calibration	√	Target Analyte List
√	Initial Calibration Verification	2	Compound Quantitation
1	Continuing Calibration	√	Compound Identification
2	Method Blank Analysis	NA	Spectral Match (Stage 4 Only)
√	Surrogate Compound Recovery	NA	Calculation Verification (Stage 4 only)

√ **Stated method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.**

1 **Quality control results are discussed below, but no data were qualified.**

2 **Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed in this Data Validation Report.**

Overall Assessment

Data were estimated based on concentrations that were greater than the calibrated linear range of the instrument. Detection limits were elevated and estimated based on method blank contamination. Results were flagged do-not-report (DNR) to indicate which result, from multiple analyses (dilutions, etc.), should not be used.

Data that have been qualified DNR should not be used for any purpose. All other data, as qualified, are acceptable for use.

Data Package Completeness

The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative. The laboratory submitted all required deliverables.

Verification of EDD to Hardcopy Data Package

Sample results and related quality control data were received in both an electronic and hardcopy format. Electronic data were verified against the laboratory report; no errors were found.

Chain-of-Custody and Sample Receipt

Sample identification (ID) numbers listed on the chain-of-custody record are consistent with the sample ID reported in the laboratory electronic data deliverable (EDD) and hardcopy data package.

Volatile samples were preserved in the field with hydrochloric acid (HCl); displayed no headspace; and were received within the advisory temperature range of 2° to 6°C (Analytical Quality Assurance Plan (AQAP, Section 3.1).

Holding Times

Samples were analyzed within the holding time specified in the Analytical AQAP, Section 3.1, and documented in the Validation Criteria Table.

Instrument Tuning

Instrument tuning was performed at the required frequency and met all criteria as specified in EPA SW846 Method 8260.

Initial Calibration

Initial calibration (ICAL) standards were analyzed at the required frequency and the relative standard deviation (%RSD) values were within the control limits specified in the AQAP, Table 6.1c and documented in the Validation Criteria Table.

The analyte tertiary butanol was calibrated using a four point curve. As the reporting limit for non-detected results were elevated to the lowest concentration of the calibration curve and as the %RSD value was acceptable, no further action was taken.

Initial Calibration Verification

Initial calibration verification (ICV) standards were analyzed required frequency and the percent recovery (%R) values were within the control limits specified in the AQAP, Table 6.1c and documented in the Validation Criteria Table. The ICV was a separate standard from a second source.

Continuing Calibration

With the exceptions noted below, continuing calibration (CCAL) standards were analyzed at the required frequency and the percent difference (%D) values were within the control limits specified in the AQAP, Table 6.1c and documented in the Validation Criteria Table.

CCAL Date	Instrument ID	Analyte	%D	Bias
5/23/2010 11:37	VOA4	1-decene	-36.1	High
5/24/2010 10:58	VOA4	nonane	-33.7	High
		1-decene	-39.0	High
		decane	-36.1	High
		1-methyl-4-propylbenzene	-28.6	High
		n-butylbenzene	-33.0	High
		1-methyl-2-propylbenzene	-25.6	High
		pentylbenzene	-32.2	High
		1,2,4,5-tetramethylbenzene	-26.3	High
		naphthalene	-25.4	High
5/26/10 02:43	VOA4	1-decene	-36.5	High

If the %D values listed above indicate a potential high bias, only the positive results in associated samples are estimated (J-5B).

In the following instances, the laboratory did not meet the MQO stated in the AQAP. In all cases, since the outliers indicated a potential high bias and the compounds were not detected in the associated sample analyses, the laboratory did not take any corrective action.

- In the CCAL analyzed on 5/23/2010, the %D value for one compound was greater than 35%.
- In the CCAL analyzed on 5/24/2010, the %D values for more than 10% of the compounds were greater than 25%. The %D values for two compounds were greater than 35%.
- In the CCAL analyzed on 5/26/2010, the %D value for one compound was greater than 35%.

Method Blank Analysis

To assess the impact of each blank contaminant on the reported sample results, two action levels are established at two (2x) and five times (5x) the concentration reported in the blank. If a contaminant is reported in an associated field sample and the concentration is less than the lower (2x) action level, the result is qualified as not detected (U-7) at the reported concentration. If a contaminant is reported in an associated field sample and the concentration is less than the higher (5x) action level, the result is estimated (J-7). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the higher action level, or for non-detected results.

Method blanks were analyzed at the appropriate frequency. Various target analytes were detected in the method blanks, however only the following analytes required qualification in one or more samples.

Method Blank ID	Analyte	Samples Qualified
VO52110B02	dodecane	JF.REF.SURF.W.20100510.N004+N005 Duplicate
VO52310B02	2-methylnaphthalene	JF.4KM.SURF.VW.20100512.N111+110

Surrogate Compound Recovery

All the percent recovery (%R) values for surrogates were within the control limits of 70 – 130%.

Sample Duplicate Analysis

At least one sample from each analytical batch (of 20 or fewer samples) was analyzed in duplicate. Where analyte concentrations in the parent sample were greater than the quantitation limit (QL), the relative percent difference (RPD) value was calculated.

Parent Sample: JF.REF.SURF.W.20100510.N004+N005

Duplicate Sample: Same

Parent Sample: JF.8KM.300FUZZ.WV.20100512.N054+053

Duplicate Sample: Same

RPD values were less than the control limit of $\leq 30\%$ for all compounds with concentrations greater than the quantitation limit.

Blank Spike/Blank Spike Duplicate Sample Analyses

One set of blank spike/blank spike duplicate (BS/BSD) samples (for each analytical batch of 20 or fewer samples) was extracted and analyzed. The percent recovery (%R) and relative percent difference (RPD) values were calculated and evaluated.

All the %R values were within the criteria of 50% – 130%. All RPD values were less than the control limit of $\leq 30\%$.

Reference Material Analysis

An aliquot of Gasoline Reference Oil LD-7 was analyzed with each set of samples. For the analysis performed on 5/23/10 (VW052310LD702), the 1-methylnaphthalene %R value was less than the 65% lower control limit, at 64%. Based on the potential low bias, the positive results and detection limits in all associated samples are estimated (J/UJ-12).

All other recovery values were within the laboratory defined criteria of 65% – 135%.

Internal Standards

All the percent recovery (%R) values for internal standards (IS) were within the control limits of 50 – 200% of the area in the associated CCAL.

Compound Quantitation

The laboratory applied a J-flag to all results between the quantitation limit (QL) and the method detection limit (MDL).

During validation, reported results less than the MDL were qualified as “found” (F).

The hexane concentrations in Samples JF.REF.MIX.WV.20100511.N029+N030 and JF.4KM.150.WV.20100512.N103+102, and the hexane and methylcyclopentane concentrations in Sample JF.4KM.360.WV.20100512.N095+N094 were greater than the calibrated linear range of the instrument. These samples were re-analyzed at a dilution (5x); however, because the vials used for the dilution analyses had previously been used for screening, headspace was present. The results in the dilutions were significantly lower than the original concentrations. Because these samples were compromised by headspace, all results from the dilution analysis were qualified as do-not-report (DNR-11). The over-calibration results in the original analyses were estimated (J-20).

Attachment 1: Sample Index - SDG 1005011
P.I.A.N.O. Volatile Organic Compounds

Sample ID	Lab ID	Date Collected
JF.REF.SURF.W.20100510.N004+N005	1005011-01	5/10/10
JF.REF.BLANK.DIV.20100510.N011+N012	1005011-02	5/10/10
JF.REF.DEEP.WR.20100511.N013+N014	1005011-03	5/11/10
JF.REF.MID.WV.20100511.N021+N022	1005011-04	5/11/10
JF.REF.MIX.WV.20100511.N029+N030	1005011-05	5/11/10
JF.REF.BLANK.DIWV.20100511.N038+037	1005011-06	5/11/10
JF.8KM.DEEP.WV.20100512.N046+045	1005011-07	5/12/10
JF.REF.SURF.WV.20100511.N025+N026	1005011-08	5/11/10
JF.8KM.300FUZZ.WV.20100512.N054+053	1005011-09	5/12/10
JF.8KM.MID.WV.20100512.N049+N050	1005011-10	5/12/10
JF.4KM.DEEP.WV.20100512.N086+N087	1005011-11	5/12/10
JF.4KM.150.WV.20100512.N103+102	1005011-12	5/12/10
JF.4KM.SURF.WV.20100512.N111+110	1005011-13	5/12/10
JF.4KM.360.WV.20100512.N095+N094	1005011-14	5/12/10
JF.8KM.MIX45.WV.20100512.N062+061	1005011-15	5/12/10
JF.REF.MIX.WV.20100511.N029+N030	1005011-05E	5/11/10
JF.4KM.150.WV.20100512.N103+102	1005011-12E	5/12/10
JF.4KM.360.WV.20100512.N095+N094	1005011-14E	5/12/10